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NOZZLE FOR SYNTHETIC FIBER SPINNING AND MANUFACTURING METHOD THEREFOR
[Kagosenboishi yo nozuru oyobi sono seizo hoho]

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Specification /439*

1. Title of the Invention

Nozzle For Synthetic Fiber Spinning and Manufacturing Method Therefor

2. Claims

(1) A nozzle for spinning synthetic fibers, wherein a plurality of nozzle openings are installed in a stainless steel plate containing Ti, and wherein at least the inside peripheral surfaces of the nozzle openings are heat treated to precipitate TiC and are coated with a corrosion and wear resistance material.

(2) A method for manufacturing a nozzle for spinning synthetic fibers, wherein a stainless steel plate containing Ti is worked and hardened, and wherein nozzles are formed in the resulting stainless steel plate, heat treated to precipitate TiC and then coated with a corrosion and wear resistance material, or the nozzles are formed, coated with a corrosion and wear resistance material and then heat treated to precipitate TiC, or nozzles are formed and coated with a corrosion and wear resistance material at a temperature that causes TiC to precipitate on the surface.

3. Detailed Description of the Invention

(Industrial Field of Application)

[01] The present invention relates to a nozzle for synthetic fiber spinning used to manufacture synthetic chemical fibers and a manufacturing method for this nozzle.

*Numbers in the margin indicate pagination in the foreign text.

(Prior Art)

[02] A nozzle for synthetic fiber spinning is one means used to manufacture synthetic chemical fibers. When synthetic chemical fibers are manufactured, a highly corrosive solution is discharged from the nozzles at high speeds. This makes the tip of the nozzle for synthetic fiber spinning susceptible to corrosion. Because fiber quality declines when the nozzles become misshapen, corrosion-resistant nozzles for synthetic fiber spinning with strength above a predetermined level are desired.

[03] These nozzles for synthetic fiber spinning are usually made of corrosion-resistant stainless steel such as SUS316, and the nozzle portion is worked and hardened to make it stronger. The surface of stainless steel such as SUS316 can also be coated with a corrosion and wear-resistant material to improve corrosion resistance and wear resistance. /440

[04] A stainless steel containing TiC can also be work-hardened and then heat treated to cause TiC surface precipitation.

[05] Stainless steel containing Ti can be heat treated to precipitate TiC between the stainless steel and a coating film in order to improve the adhesion of the coating film to the stainless steel.

(Problem Solved by the Invention)

[06] Unfortunately, when stainless steel, such as SUS316, is coated with a corrosion and wear resistant coating, the difference in thermal expansion coefficients between the stainless steel and the

coating film causes the coating film to peel off after it is heated and allowed to cool. This means the nozzle has insufficient corrosion and wear resistance.

[07] The purpose of the present invention is to solve this problem by providing a nozzle for spinning synthetic fibers and a manufacturing method for this nozzle which is strong and to which the corrosion and wear resistant coating adheres well.

(Means of Solving the Problem)

[08] In order to achieve this purpose, the present invention is a nozzle for spinning synthetic fibers, wherein a plurality of nozzle openings are installed in a stainless steel plate containing Ti, and wherein at least the inside peripheral surfaces of the nozzle openings are heat treated to precipitate TiC and are coated with a corrosion and wear resistance material.

[09] The present invention is also a method for manufacturing a nozzle for spinning synthetic fibers, wherein a stainless steel plate containing Ti is worked and hardened, and wherein nozzles are formed in the resulting stainless steel plate, heat treated to precipitate TiC and then coated with a corrosion and wear resistance material, or the nozzles are formed, coated with a corrosion and wear resistance material and then heat treated to precipitate TiC, or nozzles are formed and coated with a corrosion and wear resistance material at a temperature that causes TiC to precipitate on the surface.

[10] The heat treatment for TiC surface precipitation should be performed after the corrosion and wear resistant coating or during the

corrosion and wear resistant coating. When the TiC on the surface of the stainless steel is thick during heat treatment for TiC surface precipitation, the atoms are readily dispersed between the surface of the stainless steel and the coating film, forming a highly adhesive intermediate layer between them.

[11] Preferred corrosion and wear resistant materials include TiC, TiN, TiB_2, B_4C, BN and SiC.

[12] Methods that can be used to coat the corrosion and wear resistant material include various CVD and PVD methods such as heat CVD, plasma CVD, laser CVD, ion plating and sputtering.

[13] The heat treatment temperature for TiC surface precipitation should be set between 700 and 900°C. The TiC readily precipitates on the surface of stainless steel in this temperature range, and the precipitated TiC reduces the thermal stress on the coating film, making the coating film less likely to peel off.

[14] The heat treatment should be performed for 1 to 24 hours in a high vacuum of 10^{-3} Torr or less.

(Effect of the Invention)

[15] There were concerns that the heat treatment for TiC surface precipitation, which improves the adhesion between the stainless steel and the coating film, would cause a softening of the work-hardened layer in the nozzle for synthetic fiber spinning obtained in this manner. However, it was clear from test results that the surface retained sufficient surface hardness and that the nozzle was very strong.

[16] Therefore, even though a highly corrosive liquid is discharged from the nozzle at high speed during the manufacture of synthetic chemical fibers, the nozzle is not likely to become corroded or to experience wear. In other words, the nozzle is not likely to become misshapen. As a result, synthetic fibers with the desired level of quality can be manufactured.

(Working Examples)

[17] The following are working examples of the present invention.

[18] First, the manufacturing method for a synthetic fiber spinning nozzle will be explained with reference to a working example. The nozzle main body 1 has a round recess as shown in FIG 1. This is made of stainless steel SUS321 ($C \leq 0.08\%$, $Ti \geq 5 \times C\%$). The nozzle main body 1 is worked and hardened, and the surface is half processed. A plurality of nozzle holes 2 is formed in the nozzle main body 1. /441

[19] Next, the nozzle main body 1 (hereinafter referred to as the sample) is placed in the CVD device shown in FIG 1 and the sample surface is coated with TiN.

[20] In the CVD device, a cylindrical quartz reaction tube 5 is installed between round flanges 3 and 4. A gas input port 6 is formed in the upper flange 4 in FIG 2 to allow the reaction gas to enter the reaction tube 5, and a gas output port 7 is formed in the lower flange 3 in FIG 2 to allow the gas inside the reaction tube 5 to be discharged.

[21] A cylindrical sample platform 8 is installed inside the reaction tube 5. A coil heater 10 heated by a direct current power

source 9 is installed inside the sample platform 8, and a thermocouple 11 for detecting the temperature inside the reaction tube 5 is also installed. A high-frequency oscillating power source 12 causes plasma to fly between electrode 13 and electrode 14, and corrosion and wear resistant TiN coats the surface of the sample (the nozzle main body 1) placed on the sample platform 8.

[22] In this working example, the CVD device has the following settings. The frequency of the high-frequency oscillating power source 12 is 13.56 MHz, the temperature is 500°C, the pressure is 0.5-5 Torr, the coating time is one hour, the N_2 flow rate in the reaction gas is 40 mL/minute, the H_2 flow rate in the reaction gas is 70 mL/minute, and the TiC_4 flow rate is 0.4 mL/minute. A TiN film is formed on the surface of the sample 1 under these conditions (see FIG 3). Here, the thickness of the TiN film (coating film) 15 is 0.5-1 μm.

[23] Next, vacuum heat treatment is performed on this sample 1 for one hour at a temperature of 800°C and under pressure of 0.001 Torr.

[24] The adhesion and surface hardness of the coating film on the resulting sample were tested. In Table 1, the adhesion and surface hardness of the coating film were tested on SUS321 stainless steel (for the present invention) and SUS316 stainless steel (for the comparative example). Tests were performed for three surface processing methods: (1) CVD, (2) heat treatment followed by CVD, and (3) CVD followed by heat treatment. The adhesion and hardness units, respectively, were N (newtons) and Hv (Vickers hardness).

Table 1

		(1) CVD	(2) Heat → CVD	(3) CVD → Heat
SUS321 (Invention)	Density (N)	82	120	130
	Hardness (Hv)	500	422	459
SUS316 (Comparison)	Density (N)	78	58	71
	Hardness (Hv)	500	150	190

[25] From the results in Table 1, it is clear that the adhesion and hardness results from surface treatments (2) and (3) performed on SUS321 were higher than those for SUS316. In other words, the adhesion and surface hardness were better. The adhesion and surface hardness from method (3) obtained better results than method (2) when the heat treatment was performed after CVD.

4. Brief Explanation of the Drawings

FIG 1 is a cross-sectional view of the nozzle for synthetic fiber spinning in a working example of the present invention. FIG 2 is a simplified configurational view of a CVD device using the working example of the present invention. FIG 3 is a cross-sectional view of the nozzle hole in a nozzle manufactured using the method in a working example of the present invention.

1 ... Nozzle Main Body, 2 ... Nozzle Hole, 3, 4 ... Flanges, 5 ... Reaction Tube, 6 ... Gas Input Route, 7 ... Gas Output Route, 8 ... Sample Platform, 10 ... Coil Heater, 13, 14 ... Electrodes

FIG 1

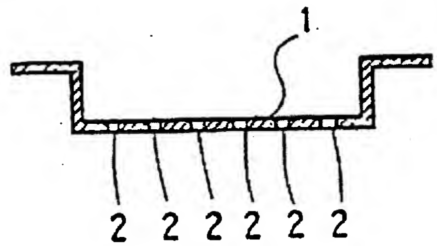

FIG 2

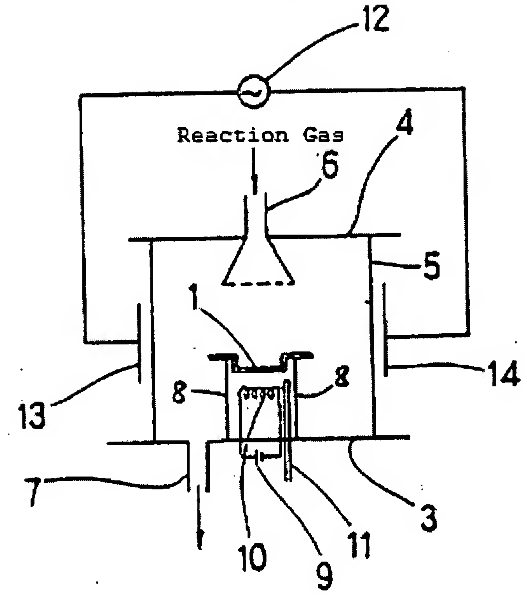

FIG 3